

PATENT ABSTRACTS OF JAPAN

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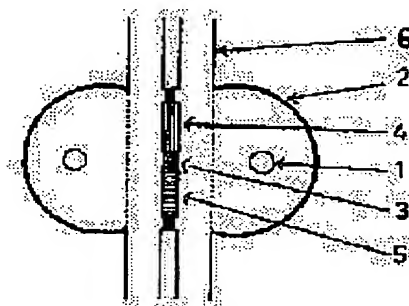
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(54) FLOATING ZONE MELTING DEVICE

(57)Abstract:

PURPOSE: To greatly improve the temp. distribution within a horizontal plane and to enable the growth of a good-quality large-sized single crystal by changing the shape of a reflection mirror of ellipsoidal surface of revolution in a radiation concentration heater of the optical floating zone melting device into a specific shape.

CONSTITUTION: An IR lamp 1 is provided on one focus of the reflection mirror 2 of ellipsoidal surface of revolution having the reflection surface on its inside surface and a melting part 3 on the other focus. The radiations reflected from the reflection surface are concentrated to this melting part to melt the part. The reflection mirror 2 of the such heater is made to the shape in which the sectional shape has the shape of ellipsoidal surface of revolution and the entire part has the annular shape. After a raw material bar 4, a seed crystal 5 and a quartz mirror 6 are set, the lamp 1 is lighted and while the bar 4 and the seed crystal 4 are rotated, the voltage impressed to the lamp is gradually increased to melt the front end of the raw material bar 4. The bar 4 and the seed crystal 5 are thereafter brought near to each other until both are joined via the melting part 3. The bar 4 and the seed crystal 6 are slowly moved downward at a prescribed speed while the voltage impressed to the lamp is adjusted, by which the bar-shaped single crystal is grown.



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CLAIMS

[Claim(s)]

[Claim 1] It is optical suspension zone melting equipment characterized by forming an infrared lamp in one focus of the ellipsoid-of-revolution reflecting mirror which made the inside the reflector, and for an ellipsoid-of-revolution reflecting mirror having an ellipsoid-of-revolution configuration as the cross section in the radiant ray centralized heating apparatus of the method which concentrates and heats the radiant ray reflected from the reflector on the focus of another side, and the whole consisting of a special configuration which becomes ring-like.

[Claim 2] Suspension zone melting equipment of claim 1 characterized by making it the structure where an ellipsoid-of-revolution reflecting mirror can be divided into two upper and lower sides.

[Claim 3] Suspension zone melting equipment of claim 1 which carries out the description of having added the device which divides into three or more pieces perpendicularly the ellipsoid-of-revolution reflecting mirror put on the horizontal plane, and can move the secondary focus of the parting line of each ellipsoid-of-revolution reflecting mirror up and down.

[Claim 4] Suspension zone melting equipment of claim 1 characterized by using what fabricated single track or this for the filament spirally in the filament configuration of the halogen lamp to be used.

[Claim 5] Suspension zone melting equipment [claim 6] of claim 1 characterized by having the device which introduces cooling air into the interior of an ellipsoid-of-revolution reflecting mirror Suspension zone melting equipment of claim 1 characterized by having the device which introduces cooling air into the interior of an ellipsoid-of-revolution reflecting mirror.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]**[0001]**

[Industrial Application] This invention concentrates the infrared radiation emitted from infrared lamps, such as a halogen lamp, as that radiant ray using an ellipsoid-of-revolution reflecting mirror, acquires an elevated temperature, and relates to the so-called radiant ray centralized heating apparatus useful although it is used for training of a single crystal, or research of a phase equilibrium.

[0002]

[Description of the Prior Art] Conventionally, it sets in fields, such as training of a single crystal, and research of a phase equilibrium. Radiant ray centralized heating apparatus is known well. This radiant ray centralized heating apparatus (1) (2) controlled atmospheres can be chosen [that melting of a sample can be performed without using crucible,] as arbitration, (3) Single crystal growth of various presentations can be easily performed using a floating zone method, (4) — that phase equilibrium research by the suspension band cooling—slowly method can be done, and (5) — with comparatively little power, there are advantages, like high temperature is obtained easily and it is widely used for training of a single crystal, the research equipment of a phase equilibrium, etc. increasingly.

[0003] Moreover, although new oxides superconductors are discovered recently and research is done briskly, in order to clarify the true character of the matter also in this case, training of a single crystal is desired, and even if it succeeds in large-sized single crystal growth in some matter using suspension zone melting equipment, it is. However, although training of an yttrium and a series of oxides—superconductors single crystals containing a bismuth is made difficult and the minute crystal is compounded, about the large-sized single crystal growth of extent which can be equal to full-scale physical-properties measurement, the example of a success does not still exist.

[0004] Conventional infrared intensive heating type suspension zone melting equipment There is no piece and two-piece (there are also four examples depending on case) use of the ellipsoid-of-revolution reflecting mirror is carried out. In bringing together the light of a halogen lamp or a xenon lamp in a focal location using this, acquiring an elevated temperature, making single crystal growth etc. perform and using one ellipsoid-of-revolution reflecting mirror It is designed so that a lamp may be formed in the location of one of the two of two foci of an ellipsoid-of-revolution reflecting mirror and the sample by which melting should be carried out may be prepared in the remaining focal location. As well as one case when using two ellipsoid-of-revolution reflecting mirrors, it is arranged fundamentally, but in this case, by making one point share the focus in which a sample should be prepared, the direction of a major axis of two ellipsoid-of-revolution reflecting mirrors is arranged on the same line, and the light of two lamps is brought together in the same location, and it is designed so that melting of a sample may be performed.

[0005] In the conventional suspension zone melting equipment designed as mentioned above, condensing, comparatively high effectiveness can be attained, and since there is an advantage from which an elevated temperature is acquired comparatively easily, it has been used for many oxide systems or research of an intermetallic compound. However, in above suspension zone melting equipment, on the relation which uses the ellipsoid-of-revolution reflecting mirror, although the temperature distribution within the horizontal plane of a sample should become fixed theoretically, there are a size effect of the filament of a lamp, a size effect of the lamp itself, etc. in fact, and the temperature distribution within the most important horizontal plane do not become fixed for single crystal growth. For this reason, rotation is added and the actual condition is aiming at the improvement of temperature distribution. And in the case of the system for which the temperature distribution controlled very strictly are made to need like [in the case of the above-mentioned oxides superconductors etc.], even if it added rotation with the heterogeneity of these temperature distribution, raising a good large-sized single crystal had the fault of being very difficult.

[0006] This invention tends to solve the trouble of the above conventional optical suspension zone melting equipments, and aims at offering the optical new suspension zone melting equipment which can improve the temperature distribution within a horizontal plane sharply.

[0007]

[Means for Solving the Problem] This invention changes the configuration of an ellipsoid-of-revolution reflecting mirror as what solves the above-mentioned technical problem. As the temperature distribution within a horizontal plane can be improved, an infrared lamp is formed in one focus of the ellipsoid-of-revolution reflecting mirror which made the inside the reflector. In the radiant ray centralized heating

apparatus of the method which concentrates and heats the radiant ray reflected from the reflector on the focus of another side. The optical suspension zone melting equipment with which an ellipsoid-of-revolution reflecting mirror is characterized by having the configuration of an ellipsoid of revolution as the cross section, and the whole consisting of a special configuration which becomes ring-like is offered.

[0008] Moreover, this invention is also what added amelioration in the configuration of those details further. Hereafter, along with the attached drawing, the equipment of this invention is explained in more detail.

[0009]

[Example] Drawing 1 and drawing 2 are the front views and side elevations having shown the concept of the infrared intensive heating type suspension zone melting equipment by this invention, and drawing 3 is the enlarged drawing of the principal part. In this drawing 1 and drawing 2, 1 is the point that an ellipsoid reflecting mirror concentrates a halogen lamp and 2, and the light of a lamp concentrates 3, and is equivalent to the fusion zone of a sample. For a raw material rod supporter and 8, as for a raw material rod support shaft mechanical component and 10, a training crystal supporter and 9 are [the crystal with which a raw material rod and 5 were raised for 4, the transparence quartz tube with which 6 forms a sample room, and 7 / a training crystal support shaft mechanical component and 11] equipment stands. In this equipment, after setting the raw material rod 4 to a raw material rod supporter, setting a seed crystal 5 to a training crystal supporter first and setting a quartz tube 6, a lamp 1 is turned on, giving rotation to a raw material rod and a seed rod by the up-and-down mechanical components 9 and 10, the applied voltage to a lamp is raised gradually and the point of the raw material rod 4 is melted. If the tip of the raw material rod 4 melts, will operate driving gears 9 and 10, a raw material rod and a seed rod will be made to approach, and both will be joined through a fusion zone. The fusion zone is held so that it may not fall with the surface tension. If a raw material rod and a seed rod are caudad moved at the rate of slowly predetermined with the same rate using the up-and-down mechanical components 9 and 10, adjusting lamp applied voltage, training of the dissolution of a raw material rod and the crystal of a up to [a seed rod] will be continued, and training of a cylindrical single crystal will be performed.

[0010] As for a crystal, training and the dissolution will be repeated by turns, and a good single crystal is not raised as a part with uneven temperature will be passed even if it rotates the training crystal drive shaft 10 how much if the temperature distribution within a horizontal plane are bad like conventional equipment at this time. However, according to this invention, as it expands also to drawing 3 and was shown in the radiant ray centralized heating apparatus of the method which concentrates and heats the radiant ray which formed the infrared lamp 1 in one focus of the ellipsoid-of-revolution reflecting mirror 2 which made the inside the reflector, formed the fusion zone 3 on the focus of another side, and was reflected in this from the reflector. Since the ellipsoid-of-revolution reflecting mirror 2 has an ellipsoid-of-revolution configuration as the cross section and the whole is considering as the special configuration which becomes ring-like, the temperature distribution within a horizontal plane are almost ideal about single crystal growth, and, thereby, a good single crystal can be raised.

[0011] furthermore, since the condensing effectiveness of a halogen lamp boils markedly other one of the descriptions of the new suspension zone melting equipment by this invention, and improves, therefore the temperature gradient of a heated object becomes steep, it is the advantage of being hard coming to generate the fall of melt made the most important for training of the diameter single crystal of macrostomia. Since the conventional floating zone melting method was an approach of making the formed melt holding in the sample itself, the amount of the melt which can be held to stability by the correlation of the surface tension of melt and the specific gravity of melt will be restricted, and training of the diameter single crystal of macrostomia had the fault by which difficulty is accompanied. However, the large-sized single crystal of the diameter of macrostomia can be raised now by using the equipment of this invention farther than before. The activity as equipment for production also spreads.

[0012]

[Effect of the Invention] By this invention, it becomes training of a good single crystal raisable [the diameter single crystal of macrostomia] as explained in detail above.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is the front view having shown an example of this invention.

[Drawing 2] It is a side elevation corresponding to drawing 1 .

[Drawing 3] It is the important section enlarged drawing of the equipment of this invention.

[Description of Notations]

- 1 Infrared Lamp
- 2 Ellipsoid Reflecting Mirror
- 3 Fusion Zone
- 4 Raw Material Rod
- 5 Seed Crystal, Training Crystal
- 6 Transparence Quartz Tube
- 7 Eight Shaft supporter
- 9 Ten Shaft mechanical component
- 11 Equipment Stand

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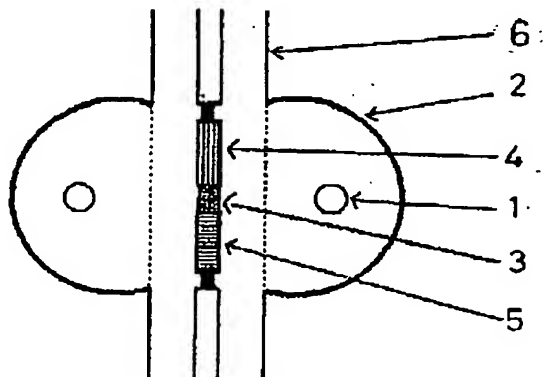
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(54)【発明の名称】 浮遊帯域溶融装置

(57)【要約】 (修正有)

【構成】 内面を反射面とした回転楕円面反射鏡の一方の焦点に赤外線ランプを設け、他方の焦点上に反射面から反射した輻射線を集中して加熱する方式の輻射線集中加熱装置において、回転楕円面反射鏡が、その断面として回転楕円面反射鏡の形状を有し、かつ、全体はリング状となる特殊形状からなることを特徴とする光学式浮遊帯域溶融装置。

【効果】 良質の単結晶の育成と、大口径単結晶の育成が可能となる。



【特許請求の範囲】

【請求項1】 内面を反射面とした回転楕円面反射鏡の一方の焦点に赤外線ランプを設け、他方の焦点上に反射面から反射した輻射線を集中して加熱する方式の輻射線集中加熱装置において、回転楕円面反射鏡が、その断面として回転楕円面形状を有し、かつ、全体はリング状となる特殊形状からなることを特徴とする光学式浮遊帯域溶融装置。

【請求項2】 回転楕円面反射鏡を上下二つに分割できる構造にしたことを特徴とする請求項1の浮遊帯域溶融装置。

【請求項3】 水平面に置かれた回転楕円面反射鏡を縦に3個以上に分割し、それぞれの回転楕円面反射鏡の分割線の第二焦点を上下に移動できる機構を付加したことを特徴する請求項1の浮遊帯域溶融装置。

【請求項4】 使用するハロゲンランプのフィラメント形状において、フィラメントを単線もしくはこれを螺旋状に成形したものを使用することを特徴とする請求項1の浮遊帯域溶融装置。

【請求項5】 回転楕円面反射鏡内部に冷却空気を導入する機構を有することを特徴とする請求項1の浮遊帯域溶融装置

【請求項6】 回転楕円面反射鏡内部に冷却空気を導入する機構を有することを特徴とする請求項1の浮遊帯域溶融装置。

【発明の詳細な説明】

【0001】

【産業上の利用分野】この発明は、ハロゲンランプ等の赤外線ランプから発せられる赤外線を回転楕円面反射鏡を用いてその輻射線として集中し、高温を得て、単結晶の育成、もしくは、相平衡の研究等に使用するのに有用な、いわゆる輻射線集中加熱装置に関するものである。

【0002】

【従来の技術とその課題】従来より、単結晶の育成、相平衡の研究等の分野においては、輻射線集中加熱装置がよく知られており、この輻射線集中加熱装置は、(1)坩堝を使用せずに、試料の溶融が行えること、(2)雰囲気ガスを任意に選べること、(3)浮遊帯域法を利用して、種々の組成の単結晶育成が容易に行えること、(4)浮遊帯域徐冷法による相平衡研究が行えること、(5)比較的少ない電力で高温度が容易に得られること、等の利点があり、単結晶の育成、相平衡の研究装置等に広く利用されるようになってきている。

【0003】また、最近、新しい酸化物超電導体が発見され、研究が盛んに行われているが、この場合にも物質の本性を明らかにするために単結晶の育成が望まれており、浮遊帯域溶融装置を用いて一部の物質においては大型の単結晶育成に成功している。しかしながら、イットリウム、およびビスマスを含む一連の酸化物超電導体単結晶の育成は困難とされ、微小結晶は合成されている

ものの、本格的な物性測定に耐えられる程度の大型の単結晶育成に関しては、いまだその成功例は無い。

【0004】従来の赤外線集中加熱式浮遊帯域溶融装置は、回転楕円面反射鏡を一個ないし二個（場合によっては四個の例もある）使用し、これを用いてハロゲンランプもしくはキセノンランプの光を焦点位置に集め、高温を得て単結晶育成等を行わせるものであり、回転楕円面反射鏡を一個用いる場合には、回転楕円面反射鏡の二つの焦点の片方の位置にランプを設け、残りの焦点位置に溶融されるべき試料が設けられるように設計されている。二つの回転楕円面反射鏡を用いる場合にも基本的には一つの場合と同様に配置されるが、この場合には、二つの回転楕円面反射鏡の長軸方向を同じ線上に配置し、試料が設けられるべき焦点を一点に共有させることで、二つのランプの光を同じ位置に集め、試料の溶融を行うように設計されている。

【0005】以上のように設計された従来の浮遊帯域溶融装置においては、比較的高い集光効率が達成でき、比較的高温が容易に得られる利点があるため、多くの酸化物系もしくは、金属間化合物の研究などに使用されている。しかしながら、上記の浮遊帯域溶融装置においては、回転楕円面反射鏡を用いている関係上、理論的には試料の水平面内の温度分布は一定になるはずであるが、実際にはランプのフィラメントの形状効果、ランプそのものの形状効果などがあり、単結晶育成にとって最も重要な水平面内の温度分布は一定にはならない。このため、回転を加えて温度分布の改善を図っているのが実情である。そして、上記の酸化物超電導体などの場合のように、極めて厳密に制御された温度分布を必要とさせる系の場合などでは、この温度分布の不均一性によって、たとえ回転を加えても、良質の大型単結晶を育成することは極めて困難であるという欠点があった。

【0006】この発明は、以上のような従来の光学式浮遊帯域溶融装置の問題点を解決しようとするものであって、水平面内の温度分布を大幅に改善できる新しい光学式浮遊帯域溶融装置を提供することを目的としている。

【0007】

【課題を解決するための手段】この発明は、上記の課題を解決するものとして、回転楕円面反射鏡の形状を変え、水平面内の温度分布を改善できるようにしたものであって、内面を反射面とした回転楕円面反射鏡の一方の焦点に赤外線ランプを設け、他方の焦点上に反射面から反射した輻射線を集中して加熱する方式の輻射線集中加熱装置において、回転楕円面反射鏡が、その断面として回転楕円面の形状を有し、かつ、全体はリング状となる特殊形状からなることを特徴とする光学式浮遊帯域溶融装置を提供するものである。

【0008】また、この発明は、さらにその細部の構成において改良を加えたものでもある。以下、添付した図面に沿ってこの発明の装置をさらに詳しく説明する。

【0009】

【実施例】図1および図2は、この発明による赤外線集中加熱式浮遊帯域溶融装置の概念を示した正面図および側面図であり、図3は、主要部の拡大図である。この図1および図2において、1はハロゲンランプ、2は楕円面反射鏡、3はランプの光が集中する点で、試料の溶融部に当たる。4は原料棒、5は育成された結晶、6は試料室を形成する透明石英管、7は原料棒支持部、8は育成結晶支持部、9は原料棒支持シャフト駆動部、10は育成結晶支持シャフト駆動部、11は、装置架台である。この装置において、まず原料棒支持部に原料棒4を、育成結晶支持部に種子結晶5をセットし、石英管6をセットしてからランプ1を点灯し、上下の駆動部9、10によって、原料棒、種子棒に回転を与えながら、徐々にランプへの印加電圧を上昇させ、原料棒4の先端部を溶かす。原料棒4の先端が溶けたら、駆動装置9、10を動かして原料棒と種子棒とを接近させ両者を溶融部を介して接合させる。溶融部は、その表面張力によって落下しないように保持されている。ランプ印加電圧を調節しながら上下の駆動部9、10を用いて、原料棒、種子棒を同じ速度でゆっくりと所定の速度で下方に移動させると原料棒の溶解、種子棒上への結晶の育成が継続され、棒状単結晶の育成が行われる。

【0010】この時、従来装置のように、水平面内の温度分布が悪いと、いくら育成結晶駆動シャフト10を回転させても、温度の不均一な部分を通過するにつれて、結晶は育成と溶解が交互に繰り返されることになり、良質の単結晶は育成されない。しかしながら、この発明によれば、図3にも拡大して示したように、内面を反射面とした回転楕円面反射鏡2の一方の焦点に赤外線ランプ1を設け、他方の焦点上に溶融部3を設け、これに反射面から反射した放射線を集中して加熱する方式の放射線集中加熱装置において、回転楕円面反射鏡2が、その断面として回転楕円面形状を有し、かつ、全体はリング状*

*となる特殊形状としているため、水平面内の温度分布は単結晶育成に関してほぼ理想的になっており、これにより、良質な単結晶が育成できる。

【0011】さらに、この発明による新しい浮遊帯域溶融装置の特徴の他の一つは、ハロゲンランプの集光効率が格段に向上し、そのために被加熱物の温度勾配が急峻となるために、大口径単結晶の育成にとって最も肝要であるとされる融液の落下が生じ難くなるという利点である。従来の浮遊帯域溶融法は、形成された融液を試料自身に保持させる方法であるから、融液の表面張力と融液の比重の相関関係によって安定に保持できる融液の量は制限されてしまい、大口径単結晶の育成には困難が伴う欠点があった。しかしながら、この発明の装置を使用することにより、従来よりもはるかに大口径の大型単結晶が育成出来るようになる。生産用装置としての活用も広がる。

【0012】

【発明の効果】この発明により、以上詳しく説明した通り、良質の単結晶の育成と、大口径単結晶の育成が可能となる。

【図面の簡単な説明】

【図1】この発明の一例を示した正面図である。

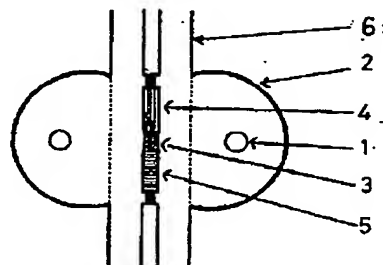
【図2】図1に対応する側面図である。

【図3】この発明の装置の要部拡大図である。

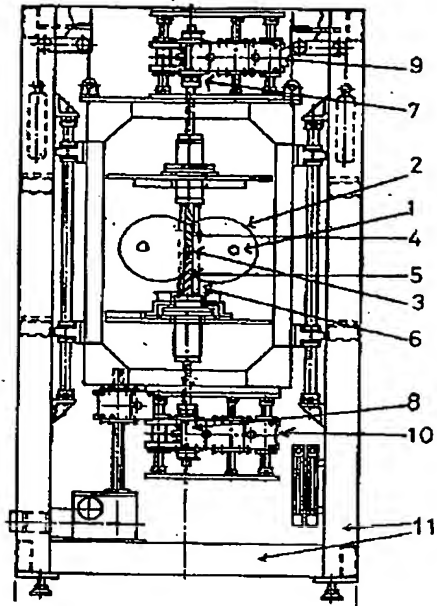
【符号の説明】

- 1 赤外線ランプ
- 2 楕円面反射鏡
- 3 溶融部
- 4 原料棒
- 5 種子結晶、育成結晶
- 6 透明石英管
- 7, 8 シャフト支持部
- 9, 10 シャフト駆動部
- 11 装置架台

【図3】



【図1】



【図2】

